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DIRECTIONS FOR DETERMINING PARTICLE SIZE OF AEROSOLS AND FINE SPRAYS

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The best method that has been found for determining the particle size
of insecticidal aerosols and fine sprays is to deposit a sample on a glass
slide and measure the particles under a high-power microscope. This method
shows the complete range of particle sizes involved. Goodhue et al. (3, 4)
used it for measuring particle sizes of aerosols deposited by settling.
This paper describes in detail the technique as it is now used.

Other, less satisfactory, methods have been devised. Gibbs (1) used
the rate of fall of the particles, and constructed a special instrument
for timing their fall. Goodhue et al. (2) used a dye in the solution and
by employing a photoelectric photometer determined the amount of deposit
per unit of time. Other workers determined by chemical analysis the rela-
tive deposition on wires of different sizes, but their results did not
clearly show the range in sizes. Instruments that pass a light beam through
an aerosol cloud and measure the polarization of light scattering at right
angles, or the number of spectra formed in the scattered light, are suitable
only for measuring particles smaller than 2 microns in diameter.

Preparation of Slides

Particles of relatively nonvolatile materials can be measured before
they evaporate. To prevent excessive spreading, filming, or coalescence,
the slide must be coated with an oleophobic substance that will cause the
individual droplets to maintain their convexity to some degree. Two of the
most satisfactory materials for this purpose proved to be a 1-percent alco-
holic solution of mannitan monolaurate, and a silicon product marketed under
the trade name Dri-film 9987. The slides are first immersed in a cleaning
solution, dried, then immersed in the oleophobic coating solution, and re-
dried. When dry the slides should be lightly polished with a soft cloth.
They may be stored in ordinary slide boxes for several days before they
are used.

Particles of volatile materials, which evaporate rapidly, cannot be
measured directly, but their size can be estimated by measuring the craters
they leave at the points of contact on slides coated with magnesium oxide
or carbon soot. It is important to apply the right thickness of coating
for the range of particle sizes anticipated. The relation between the
actual particle diameter and the central circular spot (centrum) of the
crater is illustrated in the excerpt from the report of the University of
Chicago Toxicity Laboratory, which is appended. A dye coating of polyvinyl
acetate suggested by workers in England for the same purpose proved less
satisfactory than the magnesium oxide or carbon soot.

Deposition of Particles on Slides

A sample of an aerosol or spray cloud can be deposited on a slide by impingement or by settling.

Deposition by impingement may be accomplished by moving the slide through an aerosol or spray cloud, or by moving the aerosol or spray cloud past a slide in fixed position. The velocity of movement in either case must be adjusted to the particle-size range expected. The velocity must be increased as the average particle size decreases. Since the deposition is in proportion to the particle size, compensation must be made for this factor. The Cascade and Micro Impactors developed in England, wherein an aerosol cloud is drawn through a series of orifices to vary the velocity, and impinged on a different slide at each orifice, is useful only with very small particles, mostly out of the range of insecticidal aerosols and sprays.

Deposition by settling should be limited to particle-size ranges below 20 microns in diameter. It may be accomplished by two means. An aerosol or spray cloud is released in an enclosure and allowed to settle onto slides placed on the floor or bottom. The cloud must be mixed to be uniform, and the aerosol or spray released in such a way as to prevent impingement on the sides or ceiling of the enclosure. The amount released should be small enough to prevent coalescing in the air or too heavy a deposit on the slide. Adequate time must be allowed for all the smaller particles to settle a distance equal to the height of the enclosure. Convection currents should be prevented as much as possible. A second and more rapid method is to draw the aerosol or spray cloud through an electrical precipitator in which slides have been placed. When the machine is turned on, all particles in the field are precipitated in a matter of seconds. Deposition by settling results in a slide representative of the entire range of particle sizes in the sample, with each size present in true proportion so that no adjustment or weighting is necessary.

Determination of Particle Size

After the sample of aerosol or spray has been deposited on a slide, it is placed under a microscope and the individual particles are measured with an eyepiece micrometer. A mechanical stage on the microscope is necessary. The diameter as measured on the slide is then corrected for the amount of spread that has taken place, and the diameter of the original sphere is determined.

At least 200 particles should be measured, according to DalaValle (5). The more homogeneous the aerosol or spray, the fewer particles need be counted. All particles should be counted as they are seen in the field. An accurate method is to measure all particles from one edge of a slide to the other that pass through the micrometer scale as the slide is moved by the mechanical stage. Under some conditions of impingement, particles of the smaller size groups are congregated along the margin of the slide. Measurements in such areas should be avoided.

It is sometimes useful to photograph the particles or to project them on a screen through a microscope. Better results have been obtained, however, by measuring the particles directly as seen in the microscope. It is often more convenient to measure in terms of the divisions of an eyepiece micrometer, and convert these divisions into microns after the median has been determined.

Impinged Slides.--Samples may be collected by impingement on a coated slide by waving the slide through an aerosol or spray cloud, or by drawing the aerosol or spray past a slide in fixed position, such as in a wind tunnel. The slide should be nearly perpendicular to the movement of the aerosol or spray. In either case the rate of deposition has been demonstrated to be in ratio to the square of the diameter. This rate of deposition was suggested by the Central Aerosol Laboratory of Columbia University and was based on Sell's law. To compensate for the decrease in the rate of deposition as the particle decreases in size, each diameter is multiplied by the number of particles of that size, 1/ and expressed as the percent of the total of such products. Representative data illustrating this method are given in table 1.

Table 1.--Representative count of aerosol particles impinged on microscope slides

Diameter (scale divisions)	Number of particles	Diameter times number	Percent of total of column 3	Accumulative percentage
0.5	2	1	0.2	0.2
1.0	26	26	6.3	6.5
1.5	33	49.5	11.9	18.4
2.0	82	164	39.5	57.9
2.5	34	85	20.5	78.4
3.0	17	51	12.3	90.7
3.5	4	14	3.4	94.1
4.0	5	20	4.8	98.9
4.5	1	4.5	1.1	99.9
Total	204	415.0		

1/ The diameter is used in the first power only, since the particles impinge in ratio to D^2, but the mass median diameter is computed on the basis of their volume, which is in ratio to D^3; therefore, the number of particles is multiplied by D^2/D^3, or by D.

The accumulative percentages from the last column are plotted on the arithmetic probability scale in figure 1. The 50-percent point of the line so plotted is taken as the median of the particles as they appear on the slide. In this example the 50-percent point has a value of 2 scale divisions, or 30 microns, as each division was predetermined to equal 15 microns.

A correction factor must be determined for each slide. The original spherical droplet as it is impinged on the slide becomes a convex lens, and the extent of its spread from its original shape can be calculated by determining the focal length of the lens so formed. This method is described in Porton Report 2463 (May 6), a digest of which is appended. In the example cited the correction factor is 0.40; therefore 30 microns X 0.40 gives a median particle diameter of 12.0 microns.

Settled Slides.--The median diameter of the particles collected on a slide by gravitational settling or by electric precipitation is determined by calculating the volume of each particle. The diameter of the particles is measured in microns. The volume is determined by multiplying the cube of the corrected diameter by $\pi/6$, or 0.5236. The volume of the particles of each diameter is expressed as a percentage of the total volume. Representative data illustrating this method are given in table 2. The accumulative percentages from table 2 are also plotted on the arithmetic probability scale in figure 1, and the median particle diameter is determined to be 4.05 microns.

Table 2.--A representative count of aerosol particles settled on a microscope slide. (Volume = $\pi/6$ D^3 = $0.5236 D^3$)

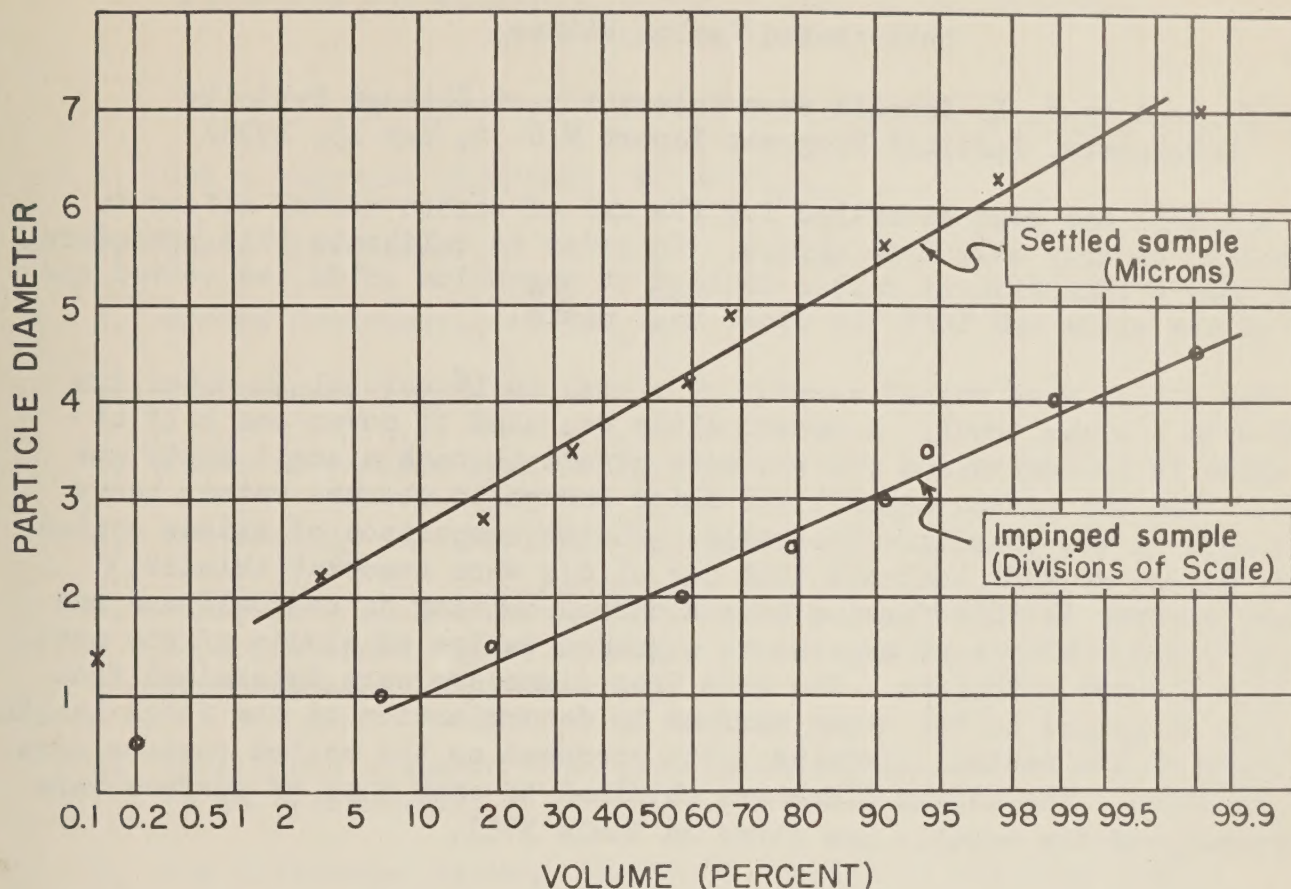| Diameter (microns)	Number of particles	Volume (microns^3)	Percent of total volume	Accumulative percentage
1.4	1	1	0.01	0.01
2.1	55	267	3.3	3.31
2.8	101	1161	14.5	17.81
3.5	50	1119	14.0	31.81
4.2	57	2211	27.7	59.51
4.9	11	677	8.5	68.01
5.6	20	1838	23.0	91.01
6.3	4	524	6.6	97.61
7.0	1	180	2.3	99.91
Totals	300	7978		

Figure 1.--Percentage of the total volume of aerosol samples below each stated particle diameter impinged and settled on microscope slides. The mass median diameter is determined from the 50-percent point. The correction factor for spread has been applied to the data for the settled slide (from table 2) but not the data for the impinged slide (table 1).

Literature Cited

(1) Gibbs, W. E.
        1924. Clouds and smokes. 240 pp. Philadelphia.

(2) Goodhue, Lyle D., and Sullivan, W. N.
        1943. Making and testing aerosols. Amer. Assoc. Adv. Sci.,
            Pub. 20: 157-162.

(3) _____, Diamond, P. T., and Riley, R. L.
        1945. Determination of particle size of liquefied gas aerosols.
            U. S. Dept. Com., O. T. S., PB 76015.

(4) _____, and Riley, R. L.
        1946. Particle-size distribution in liquefied-gas aerosols. Jour.
            Econ. Ent. 39: 223-226.

(5) DalaValle, J. M.
        1943. Micromeritics. 428 pp. New York.

(6) May, K. R.
        1942. The Cascade Impactor--an apparatus for sampling solid and
            liquid particulate clouds. Brit. Commonwealth Sci. Off.
            Porton Rpt. 2463, 7 pp.

APPENDIX

Thin-coated Carbon Slides

(Excerpt by W. R. Schmitz from University of Chicago Toxicity
Laboratory, Informal Progress Report N.S. 2, May 15, 1945)

A method has been described for the use of carbon-coated slides in
estimation of drop size from sprays. In order to calibrate this procedure,
which uses a thin film of carbon instead of magnesium oxide, we coated one
half of the slide and left the other half plain.

The slides were washed carefully, rinsed in 1% aerosol in water, and
dried with a clean towel. A second slide was used to cover one half of
the slide to be coated, and the two were passed through a small sooty gas
flame. When the carbon deposit was thick enough to obscure rather heavy
black type it was considered suitable. (Later comparison of slides coated
by Lt. Wilson at NMRI indicate that our slides were somewhat thinner.)
Slides prepared in this fashion have a carbon coating on one half and one
half is clean. They were exposed to a graded series of clouds of the non-
volatile dibutyl phthalate. The true drop diameters were determined from
the lens diameters on the clean surface by determination of the focal length.
The sizes of the central circular spots produced on the coated surface were
also measured. The median diameters obtained on each type of surface were
determined, and the results are given in Table XVII.

Table XVII

Slide No.	True mass median diameter	Ratio $\dfrac{\text{drop diameter}}{\text{diameter of centrum}}$
1	4.48	0.71
2	4.63	0.89
3	9.09	0.81
4	10.5	0.95
5	17.9	0.70
6	18.8	0.74
7	20.8	0.67
8	49.0	0.67
9	51.5	0.54

There is ample evidence of marked variation in the ratio of drop
diameter to centrum diameter of different slides, and the relation of this
ratio to increasing diameter (MMD) is not regular. We have found no simple
method, applicable to field operations, which will ensure uniformly thick
coatings on all slides. Furthermore the best optical definition occurs
at a thickness of carbon coating which is a function of the size of the
drop. If the coating is very thin it is possible to detect drops 2 microns
in diameter but larger drops are poorly defined; conversely, small drops
are invisible on thick coatings. The diameter and appearance of the annulus
surrounding the centrum appear to be very sensitive to variations in thick-
ness of coating.

Procedure for Measuring Spread Factor of Oil Droplets
on Oleophobic Slides

(A digest of Porton Report No. 2463)

1. Use a compound high-power microscope.

2. Use a flat mirror.

3. Remove condenser.

4. Use outside light.

5. Focus on particle, and measure and record exact diameter.

6. Set reading on fine focus adjustment at zero.

7. Manipulate coarse focus adjustment and mirror until some distant object (window frame) is in as sharp a focus as possible, using the drop as a lens.

8. Then focus downward with fine focus adjustment until the drop is in clear focus.

9. The difference between the No. 6 reading of the fine focus adjustment (zero) and the No. 8 reading is the focal-length change.

10. Compute $\dfrac{f' \ \text{(focal-length change)}}{2A \ \text{(diameter of particle)}}$

> Example: The diameter of a particle covering 4 divisions in an eyepiece micrometer (1 division = 15.4 microns) would be 4 X 15.4 microns, or 61.6 microns. With a focal-length change of 206 microns, the spread factor of the particle would be 206/61.6, or 3.3.

With this factor of 3.3, the correction factor ($f'/2A$) for this drop would be 0.40 (see below for spread-factor ratios).

Spread-Factor Ratios

$\dfrac{f'}{2A}$	Correction Factor	$\dfrac{f'}{2A}$	Correction Factor	$\dfrac{f'}{2A}$	Correction Factor
1.48	0.60	2.0	0.48	4.0	0.375
1.50	0.58	2.1	0.47	4.8	0.35
1.55	0.55	2.2	0.46	5.0	0.34
1.60	0.54	2.3	0.45	5.5	0.33
1.65	0.53	2.6	0.44	6.0	0.32
1.70	0.52	2.65	0.43	6.8	0.31
1.75	0.51	2.8	0.42	7.0	0.30
1.80	0.50	3.1	0.41	8.0	0.29
1.95	0.49	3.3	0.40	9.0	0.28
				10.0	0.27